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(54) Title: METHOD FOR PRODUCING A SPUNLACE MATERIAL WITH INCREASED WET STRENGTH AND SPUNLACE MATERIAL ACCORDING TO THE METHOD

(57) Abstract

Hydro-entangled nonwoven material which, after the hydro-entanglement, is subjected to plasma or corona treatment with a view to increasing the wet strength of the material. It is believed that the surface of the fibres is modified by the treatment in such a manner that the fibre-to-fibre friction increases.

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METHOD FOR PRODUCING A SPUNLACE MATERIAL WITH INCREASED WET STRENGTH AND SPUNLACE MATERIAL ACCORDING TO THE METHOD

BACKGROUND OF THE INVENTION

The present invention relates to a method for producing a hydro-entangled nonwoven material with increased wet strength.

Hydro-entanglement or spunlacing is a method which was introduced in the 1970s, see for example Canadian patent no.841,938. The method involves forming either a dry-laid or wet-laid fibre web, whereafter the fibres are entangled by means of very fine water jets under high pressure. A plurality of rows of water jets are directed towards the fibre web which is carried on a displaceable wire. The entangled web is thereafter dried. Those fibres which are used in the material can be synthetic or regenerated staple fibres, e.g. polyester, polyamide, polypropylene, rayon and the like, pulp fibres or a mixture of pulp fibres and staple fibres. Spunlace materials can be produced to a high quality at reasonable cost and display high absorption capability. They are used *inter alia* as wiping materials for household or industrial applications, as disposable materials within health care, etc.

Spunlace material based on mixtures of pulp fibres and relatively short (< 25mm) synthetic or vegetable fibres often have good strength properties in a dry condition. The binding system in this type of material is, in a dry condition, a combination of friction between all the fibres in the material and of hydrogen bonds between the pulp fibres in the material. In water and other polar solvents, the hydrogen bonds between the pulp fibres more or less disappear and the strength of the material becomes very

dependent on the friction between the fibres in a wet condition.

This sensitivity to polar solvents can be reduced by the addition of various binders such as latex of conventional type based, for example, on ethylvinyl acetate, acrylates or styrene butadine. Wet strength resins of, for example, polyamide epichlorohydrine type also improve the strength properties of spunlace material.

Reinforcement of spunlace material with the help of various binders can result in a number of problems of more or less serious nature depending on where and how the material is to be used. Certain chemical binders have poor resistance to commonly occurring solvents, something which is a significant drawback for nonwoven material which is used in wiping cloths for cleaning together with solvents. Binder-reinforcement often creates a stiffening of the material, which can also be a significant drawback for certain applications in which a soft and drapable nonwoven material is required. Furthermore, the addition of a binder is a chemical treatment which is often less desirable from an environmental point of view.

Another method of raising the wet strength in spunlace material is by thermal bonding, which can be used where the material contains thermoplastic fibres. In such cases, the thermoplastic fibres in the material are melted after the hydro-entanglement by means of raised temperature and pressure. The drawback with this method is that the material becomes stiffer and the fused thermoplastic fibres can locally form hard regions which can score delicate surfaces during, for example, polishing. A further drawback with thermal bonding is that fibre-recycling becomes more difficult with mixed material (e.g. cellulose/ polypropylene).

It is also conceivable to increase the friction of the synthetic fibres initially in connection with the fibre production. This, however, creates problems during wet- or foam-forming where it is desirable to have the lowest possible fibre-to-fibre friction in order to maintain as even a dispersion as possible during the forming. In addition, relatively low fibre-to-fibre friction is required for the subsequent hydro-entanglement if good entanglement results are desired.

A number of new methods for chemically-physically affecting the surface of different materials have been developed during the last few years. Among these methods there can be mentioned electron radiation, ultra-violet methods and plasma methods. The advantage of these methods is that the treatment occurs in the gaseous phase and thus the material is gently treated and no subsequent drying or after-treatment is required.

Plasma is a general term for gases which comprise ions, electrons, free radicals, photons within the UV-range, molecules and atoms. Plasma is electrically neutral and is normally generated by electric discharge in which the energy source is in the form of radio or microwaves.

Plasma treatment can be said to be a further development of corona treatment and the primary difference is that corona treatment takes place at atmospheric pressure whilst so-called glow discharge in cold plasma takes place at reduced pressure. Plasma treatment can be executed in the presence of different gases depending on which result is desired.

Plasma treatment is used nowadays, for example, to provide plastic components with a coatable surface. It is also used to chemically modify the surface on fibres with an aim to increase the wettability of fibres, as well as to increase

the adherence between fibres and a filler. Plasma treatment of reinforcement fibres which are to be embedded in a thermoplastic matrix is described in US-A-5,108,780. It is believed that the effect of the plasma treatment is that free radicals are formed on the fibre or material surface. These free radicals can then react with each other, with components in the plasma phase or with molecules in the atmosphere, for example oxygen gas, as soon as the treated material is removed from the plasma reactor.

Corona treatment has long since been used to morphologically and chemically modify the surface of polymer films and in particular for the purpose of improving the adhesion of printing ink or to perforate the film. Apparatus for corona treatment is described in, for example, US-A-4,283,291. It is also known from, for example, US-A-4,535,020 and EP-A-0,483,859 to treat surface material for absorbent products such as diapers and sanitary napkins with corona at the same that the material is also treated with a surfactant to increase the liquid permeability. Thanks to the corona treatment, an improved permanent wettability is attained. In EP-A-484,930 it is disclosed that wiping cloths of, for example, meltblown material can be treated with corona to provide the material with improved permanent absorption properties during repeated use.

OBJECT OF THE INVENTION

The object of the present invention is to provide a spunlace material which displays improved strength properties particularly in a wet condition by means of an after-treatment of the material without the addition of binders or thermal bonding. This is achieved according to the invention by subjecting the material to plasma or corona treatment after the hydro-entanglement. The plasma

or corona treatment is believed to modify the surface of the fibres in such a manner that the fibre-to-fibre friction increases, something which would explain the improved strength properties of the treated material.

DESCRIPTION OF THE INVENTION

Plasma treatment has been shown to be a very effective method of modifying the parameter which is desired to be changed in the described type of nonwoven material, i.e. the fibre-to-fibre friction in a wet condition. Surface modification by corona discharge at atmospheric pressure has also been shown to provide significant increases in the wet strength of the spunlace materials in question.

The fibres making up the material can be synthetic or regenerated staple fibres, e.g. polyester, polyamide, polypropylene, rayon or the like, vegetable fibres, pulp fibres or mixtures thereof. The pulp fibres can be of chemical, mechanical, thermomechanical, chemical-mechanical or chemical-thermomechanical pulp (CTMP). Addition of mechanical, thermomechanical, chemical-mechanical or chemical-thermomechanical pulp fibres provides a material with higher bulk and improved absorption and softness, which is described in our Swedish application no.9500585-6. The strength properties are, however, worsened which is why an after-treatment to increase the strength of the material can be necessary for certain applications. Plasma or corona treatment can thus be a suitable alternative.

Examples of vegetable fibres which can be used are leaf fibres such as abaca, pinapple and phormium tenax, bast fibres such as flax, hemp and ramie and seed hair fibres such as cotton, kapok and milkweed. During the addition of such long hydrophillic vegetable fibres in wet- or foam-formed materials, it may be necessary to add a dispersion

agent, for example a mixture of 75% bis(hydro-generated tallowalkyl)dimethyl ammonium chloride and 25% propylene glycol. This is described in greater detail in Swedish application nr. 9403618-3.

A certain proportion of recycled fibres from textile waste, nonwoven waste and the like may also be included in the material. This is described in Swedish application nr. 9402804-0. Since such material has lower strength compared to materials based on virgin fibre raw material, plasma or corona treatment can be a suitable method of improving the strength properties of these materials.

During production of dry-formed spunlace materials, dry fibres are airlaid on a wire, whereafter the fibre web is subjected to hydro-entanglement. During production of wet- or foam-formed material, the fibres are dispersed in liquid or in a foamed liquid containing a foam-forming surfactant and water. One example of a suitable such foam forming method is described in Swedish application nr. 9402470-0. The fibre dispersion is drained on a wire and hydro-entangled with an energy input which may suitably lie in the range 200-800 kWh/ton. The hydro-entanglement takes place using conventional methods and equipment which is provided by machine manufacturers. Production of dry- and wet-formed spunlace material is described in, for example, CA 841,938.

The hydro-entanglement of a wet- or foam-formed fibre web can either take place in-line, i.e. immediately after the fibre web has been drained on the wire, or on a wet-formed sheet which has been dried and wound up after the forming. A plurality of such sheets can be laminated together by hydro-entanglement. It is also possible to combine dry-forming with wet- or foam-forming in such a manner that an airlaid web of, for example, synthetic fibres are entangled

together with a wet- or foam-formed paper sheet of pulp fibres, see for example CA 841,938 and EP-B-0,108,621. After the hydro-entanglement, the material is pressed and dried then wound up. The thus produced material is thereafter converted in a known manner to a suitable format and packaged.

The invention is of particularly great significance for wet- and foam-formed spunlace material where the choice of fibre length is more restricted since too long fibres are difficult to disperse in liquid or foam. The problem with sufficient wet strength is normally greater in a material which contains short fibres.

The plasma or corona treatment of the material suitably takes place on the dry material before it is wound up. By the expression "dry material" is meant a material which has a moisture content of maximum 10% by weight calculated on the total weight of the material. An example of gases which can be used during plasma treatment at reduced pressure are oxygen, nitrogen, argon, helium, ammonia, carbon tetrafluoride, carbon dioxide and organic unsaturated gases. Oxygen or nitrogen are hereby preferred. The material which is to be treated is fed through a plasma plant of commercially available type, e.g. from Centexbel. The treatment preferably takes place continuously, i.e. the material is fed continually through a vacuum chamber which contains electrodes, injection and evacuation means for the used gas, feeding means for the material and a high frequency generator.

Corona treatment can take place using commercially available equipment, e.g. Ahlbrandt System ASOH12.

Examples

Several different materials with different fibre compositions were produced by wet- or foam-forming methods with subsequent hydro-entanglement. The materials were thereafter subjected to plasma treatment at reduced pressure (0,7 mbar) in the presence of either oxygen or nitrogen gas. Alternatively, the material was subjected to corona treatment at atmospheric pressure.

Comparisons were made with reference material which had not been subjected to plasma or corona treatment. The fibres of the materials were a mixture of chemical pulp fibres and synthetic fibres. The chemical pulp fibres were bleached chemical softwood pulp. The synthetic fibres which were used were polyester 1,5 dtex x 12,7 mm, respectively polypropylene 1,7 dtex x 12 mm and 1,7 dtex x 18 mm. The hydro-entanglement took place with an energy input of about 600 kWh/ton. After the hydro-entanglement and before the plasma or corona treatment, the materials were lightly pressed and dried by through-air drying at 130°C. The properties of the materials are listed in Tables 1 and 2 below.

TABLE 1

Effect of plasma treatment at reduced pressure on several material properties of hydro-entangled nonwoven material with different fibre compositions

Batchwise treatment in laboratory equipment was executed in the presence of different gases, at different power levels and treatment times

	A-ref	A1	A2	B-ref	B1	B2	C-ref	C1	C2	C3
FORMING METHOD	wet-forming			wet-forming			foam forming			
1) % CHEMICAL PULP FIBRE	60			60			60			
2) % POLYESTER 1.5dtex * 12.7mm	40			-			-			
3) % POLYPROPYLENE 1.7dtex * 12mm	-			40			-			
4) % POLYPROPYLENE 1.7dtex * 18mm	-			-			40			
*ENTANGLEMENT ENERGY, KWh/ton	≈600			≈600			≈600			
PRESSING	light			light			light			
THROUGH AIR DRYING, °C	130			130			110			
PLASMA TREATMENT GAS	-	N2	O2	-	N2	O2	-	O2	O2	O2
PRESSURE IN PLASMA FURNACE	-	0.7 mbar	0.7 mbar	-	0.7 mbar	0.7 mbar	-	0.7 mbar	0.7 mbar	0.7 mbar
POWER	-	300 W	300 W	-	300 W	300 W	-	100 W	200 W	300 W
TREATMENT TIME	-	3 min	3 min	-	3 min	3 min	-	10 sek	2 min	3 min
5) BASIS WEIGHT, g/m^2	89.9	85.0	84.2	79.4	84.3	89.7	88.6	87.2	88.3	94.0
6) THICKNESS, µm	406	402	405	469	494	512	448	434	450	451
7) BULK, cm^3/g	4.5	4.7	4.8	5.9	5.9	5.7	5.1	5	5.1	4.8
8) TENSILE STRENGTH Cross dir., N/m	957	978	1235	470	667	711	817	947	916	952
9) WET TENS. STRENGTH Cross dir., N/m	284	1025	1087	169	513	635	245	716	759	679
10) RELATIVE WET STRENGTH (wet/dry*100), %	30	105	88	36	77	89	30	76	83	71

**) Entanglement energy calculated on added quantity of fibre.*
1) commercially available bleached chemical softwood pulp.
2) commercially available polyester fibre for wetlaid nonwoven.
3) commercially available polypropylene fibre for wetlaid nonwoven.
4) commercially available polypropylene fibre for wetlaid nonwoven.
5) SCAN-P 6:75
6) SCAN-P 47:83
7) thickness/basis weight
8) SCAN-P 38:80
9) SCAN-p 58:86
*10) wet/dry*100%*

TABLE

Effect of corona treatment at atmospheric pressure on several material properties of a foam-formed hydro-entangled nonwoven material.

Double-sided corona treatment was executed in pilot equipment on a continuous web at atmospheric pressure.

	untreated	Corona treated	
FORMING METHOD	foam forming		
% CHEMICAL PULP FIBRE	60		1)
% POLYPROPYLENE 1.7 dtex * 18mm	40		2)
*ENTANGLEMENT ENERGY, KWh/ton	∝600		
PRESSING	light		
THROUGH-AIR DRYING, °C	110		
LINE SPEED CORONA	-	10 m/min	
POWER CORONA TREATMENT	-	100 W/cm	
BASIS WEIGHT, g/m²	72.8	78.4	SCAN-P 6:75
THICKNESS, µm	391	402	SCAN-P 47:83
BULK, cm³/g	5.4	5.1	thickness/basis weight
TENSILE STRENGTH Cross dir., N/m	618	777	SCAN-P 38:80
WET TENSILE STRENGTH Cross dir., N/m	162	415	SCAN-P 58:86
RELATIVE WET STRENGTH (wet/dry*100),%	26	53	wet/dry * 100%

*) Entanglement energy calculated on added quantity of fibre.

1) commercially available bleached chemical softwood pulp.

2) commercially available polypropylene fibre for wetlaid nonwoven.

The results show that the wet tensile strength of the plasma-treated materials and corona-treated material was increased several-fold. The dry tensile strength also increased somewhat. The large increase in the wet tensile strength is thought to be due to the plasma and corona treatments modifying the surface of the fibres in such a manner that the fibre-to-fibre friction increases. Since it is precisely the wet tensile strength which has often been the problem in spunlace material, the invention offers a solution to a previously difficult-to-solve problem. The solution according to the invention further implies that the need for binders and other wet tensile strength increasing chemicals as well as thermal bonding is eliminated.

As a result of its high wet strength, the material is eminently suitable as wiping material for household use or for commercial use in workshops, industry, hospitals and other public sectors. It may also be used as disposable material within health care, e.g. surgical gowns, bed sheets and the like. It may also be used as a component in absorbent products such as sanitary napkins, panty liners, diapers, incontinence products, bedding, wound dressings, compresses and the like.

CLAIMS

1. Method for producing a hydro-entangled nonwoven material with increased wet strength, characterized in that after the hydro-entanglement, the material is subjected to plasma or corona treatment.

2. Method according to claim 1, characterized in that the plasma treatment is executed at a reduced pressure in the presence of a gas selected from the group comprising oxygen, nitrogen, argon, helium, ammonia, carbon tetrafluoride, carbon dioxide, organic unsaturated gases or mixtures thereof.

3. Method according to claim 2, characterized in that the gas is preferably oxygen or nitrogen or mixtures thereof.

4. Method according to any one of the preceding claims, characterized in that the plasma or corona treatment is carried out after the material has been subjected to drying after the hydro-entanglement.

5. Method according to any one of the preceding claims, characterized in that the hydro-entangled material is produced from a wet- or foam-formed fibre web.

6. Hydro-entangled nonwoven material with increased wet strength, characterized in that after the hydro-entanglement, the material has been plasma- or corona-treated.

7. Nonwoven material according to claim 6, characterized in that the fibres of the material are synthetic or regenerated staple fibres, for

example polyester, polyamide, polypropylene, rayon and the like, vegetable fibres, pulp fibres or mixtures thereof.

8. Nonwoven material according to claim 7, c h a r a c t e r i z e d i n that the material contains a certain proportion of recycled fibres from nonwoven waste, textile waste or the like.

9. Non-woven according to any one of claims 6-8, c h a r a c t e r i z e d i n that the material is a wet- or foam-formed spunlace material.

INTERNATIONAL SEARCH REPORT

International application No.
PCT/SE 96/00199

A. CLASSIFICATION OF SUBJECT MATTER

IPC6: D06M 10/02

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

IPC6: D06M

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

SE,DK,FI,NO classes as above

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

WPI, PIRA, TEXTILE TECHNOLOGY DIGEST

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
Y	GB 2120694 A (JOHN CHRISTOPHER ROBERTS), 7 December 1983 (07.12.83) --	1-9
Y	Dialog Information Services, File 248, PIRA, Dialog accession no. 00393001, Pira accession no. 20016482, Anon: "Delaware Operation Offers Corona Treating", Nonwovens Mark. vol., 9, no. 17, 26 Aug. 1994, p. 3 --	1-9
A	EP 0483859 A1 (KIMBERLY-CLARK CORPORATION), 6 May 1992 (06.05.92) -- --------	1-9

☐ Further documents are listed in the continuation of Box C.

☒ See patent family annex.

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INTERNATIONAL SEARCH REPORT

Information on patent family members

01/04/96

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Patent document cited in search report	Publication date	Patent family member(s)	Publication date
GB-A- 2120694	07/12/83	NONE	
EP-A1- 0483859	06/05/92	SE-T3- 0483859	
		AU-B- 646303	17/02/94
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		DE-D,T- 69111561	18/01/96
		US-A- 5112690	12/05/92